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Our Ref: M/2016/19/C1
Your Ref.: 1729 Farmhouse

10th September 2019

CERTIFICATE OF ANALYSIS OF A MORTAR AND PLASTER SAMPLE FOR DETERMINATION OF MIX COMPOSITION, GRADING ANALYSIS AND BINDER TYPE

Project Reference	:	1729 Farmhouse, Malton, North Yorkshire
Sample Description	:	S1 – Original Masonry Bedding Mortar S2 – Original Plaster Mortar.
Date Received	:	18th July 2019
CMC Sample Ref	:	SR 2740 – S1 and S2.
Method of Test	:	Determination of mix composition by acid digestion with grading of recovered aggregates. Mineralogical composition and binder type by X-Ray Diffraction, with the form of binder and fabric condition assessed from a thin section examination.

Sample

Two samples of mortar were received in CMC's Stirling laboratory on the 18th July 2019. The samples were submitted for analysis by Nigel Copsey of the Earth, Stone and Lime Company, and were identified as masonry bedding mortar and plaster. The samples were stated to have been obtained from a 1729 Limestone masonry Farmhouse in Malton, North Yorkshire.

The samples were received along with a request that they be analysed to determine the mix composition, and grading of the aggregates, with binder type determination along with examination to assess the form in which the binders were used and confirm if they contained lime as part of the binder.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2740 allocated. The client's sample reference and the locations sampled are detailed below:

CMC Sample Ref.	Client Ref	Location Sampled
SR2740 – S1	Bedding Mortar	Original Bedding Mortar, from masonry bedding, 1729 Farmhouse,
SR2740 – S2	Plaster	Original Plaster Mortar, from a 1729 Farmhouse.

Method of Test

The samples were initially photographed on receipt in the laboratory and logged, with their mass and size recorded prior to being prepared for analysis. The samples were also submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x10.

During the microscopic examination the samples were exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions to aid in the identification of the components present and to assess the condition of the mortars as received.

Following the initial examination, a representative sub-sample was obtained from each sample with these dried to a constant weight prior to being lightly ground and disaggregated in an agate mortar and pestle in preparation for determination of their mix composition, where appropriate, by acid digestion, with the particle size distribution of the aggregates determined by wash grading analysis.

To establish the binder type used in the production of the mortar in the samples, and to establish if there was lime in the samples, further sub-samples were submitted to analysis by X-ray Diffraction (XRD). This was achieved by lightly crushing and grinding a further representative sub-sample, from each sample for analysis. With the fines rich material, passing a 63µm sieve, collected and back-packed into proprietary sample holders in preparation for presentation in the X-Ray Diffractometer.

In addition to the above a petrographic thin section was prepared from each sample. This was to permit comment to be made on the fabric condition of each and to confirm the form in which any lime identified, was used in the production of the mortar.

Observations from Macro and Microscopic examination

The samples were logged on receipt with the following determined:

Sample Ref.	Client Ref.	Mass of Sample (gram)	Dimensions of Largest piece (mm)	Colour by the Munsell Soil Colour Charts
SR2740-S1	Bedding Mortar	161.3	49.3 x 41.8 x 28.3	10YR 6/6 "Brownish Yellow"
SR2740-S2	Plaster	387.0	94.5 x 550. x 42.1	10YR 6/6 "Brownish Yellow"

Sample SR2740-S1 - Original "Clay" Bedding Mortar, *c* 1729

This sample was received in the form of several fragments of a clay bound mortar along with a small quantity of unbound mortar fines. The largest intact fragment measured 49.3mm (largest dimension) and was found to be well compacted, firm and moderately hard. This fragment, along with the other intact pieces, were noted to consist of a quantity of sand grains bound in a clay matrix. However, small lime inclusions were also observed in the intact pieces, along with small fragments of unburnt limestone. The mortar pieces could be broken under moderate to moderately firm finger pressure but once disrupted the mortar could be disaggregated and powdered with relative ease. However, a number of the smaller fragments were found to be particularly friable and these may have been disrupted during sample extraction.

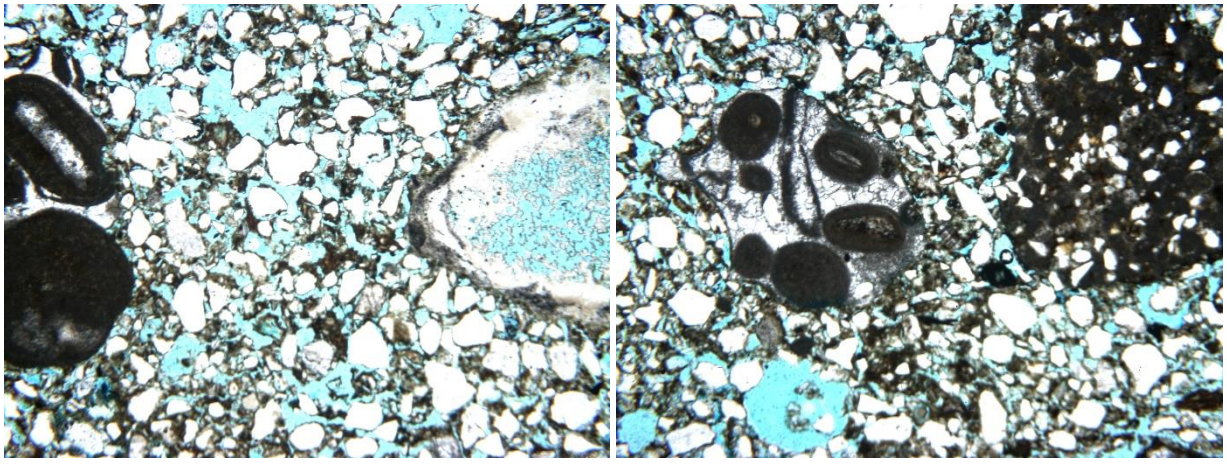
The as received moisture content was measured at 2.4% by dry mass.

From a visual examination with the aid of a stereo-binocular microscope it was noted that some of the small white 'aggregates', were limestone fragments, although lime inclusions, up to 1.4mm were also observed within the sample. Spot tests on the mortar fabric also indicated that there was acid soluble material present within the bound binder fabric, and on testing intact pieces and disaggregated mortar with a phenolphthalein indicator solution, the lime was found to be fully carbonated.

Although the aggregates displayed a mixed lithology, they are dominated by quartz with minor quartzite, weathered sandstone fragments and indeterminate rock fragments, felspar and muscovite mica were noted to be dominant in the fines fractions. Although the finer fractions were found to consist of mostly silt sized grains, a low proportion of clay was also indicated to be present.

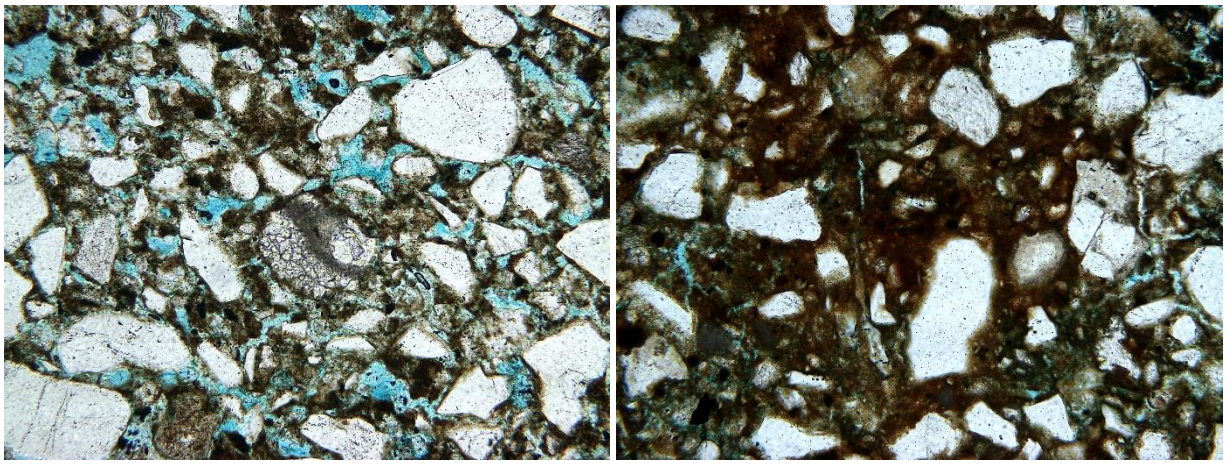
Small fragments of wood were also observed in addition to small pockets of ash material. Locally root fibres were observed in two of the pieces, with these remaining pliable and are perhaps indicative of post placement development, albeit they are not fresh and do not show evidence of continued growth.

Plates No. 1, 2 & 3: The left plate shows the condition of the mortar sample as received. The centre plate shows one of the intact fragments with limestone fragments and root fibres present in the mortar, arrowed. The right plate shows a close-up of a large limestone fragment, with the presence of limestone dust also apparent in this fragment, not observed in all pieces.



Plates No. 4 & 5: The above are images of the thin section prepared from the largest intact piece of mortar, viewed in the polarised light microscope. The left plate, viewed in plane polarised light (ppl), shows the fabric containing an abundance of fine quartz grains, from the sand (white in image) with a piece of Oolitic limestone at the left margin and a void formed from the depletion of a lime inclusion at the right margin. The aggregate particles are mostly bound in a clay and silt rich paste. The aggregates are dominated by quartz, with minor sandstone/siltstone fragments with feldspar and mica in the fines fraction. The right plate again shows a limestone fragment, centre left, which is fresh and shows no evidence of having been burned. Porosity and voids are highlighted by the blue dyed resin. The field of view in both of the above images is 2.4mm.

The mortar contains an abundance of voids which are both placement features and the result of early drying shrinkage. These have formed an abundance of very fine connecting channel ways, which along with the microporosity apparent, will ensure that the mortar is vapour permeable and relatively free draining.



Plates No. 6 & 7: The above are magnified views of the mortar fabric, with a field of view of 1.2mm. The left plate shows a sand rich area of fabric, in which small limestone fragments can be seen along with patches of fine carbonated lime intermixed with the clay and silt rich paste. With the right plate showing a lime free very dense clay rich area, where the void content is low, and the sand grains are fully encapsulated within the clay/silt rich matrix.

A modal analysis was carried out to determine the mix composition, with the results presented in the following table:

Sample Ref:	**SR2740-S1**	
Constituents	**%**	
Aggregate	**Inclusions as Binder**	**Inclusions as Aggregate**
Quartz	41.1	41.1
Limestone	3.6	3.6
Lithic Fragments	2.4	2.4
Sandstone/Siltstone	2.4	2.4
Feldspar	4.1	4.1
Mica	2.1	2.1
Opaque/Coal/Ironstone	2.4	2.4
Wood/root/straw	0.6	0.6
Lime inclusions	-	2.8
Total Aggregate	**58.7**	**61.5**
Paste (Lime/clay/silt)	38.5	38.5
Lime inclusions	2.8	-
Clinker	0	0
Secondary products	0	0
Total Binder	**41.3**	**38.5**
Total Constituents	**100.0**	**100.0**
Voids & Cracks	**15.0**	**15.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 1.4**	**1.0 : 1.6**

Table No. 1: Result of modal analysis on thin section from sample S1

The effective binder content reported in the above table is calculated on the basis that the inclusions are acting as aggregate rather than as binder and is probably a truer measure of the binder content of the mix, with regard to its performance as a mortar. Whereas, the total lime content reflects the mix at the time of mixing, including the inclusions as part of the added lime binder.

The clay to lime content, determined from the acid digestion, and corrected from the data obtained from the XRD analysis and the modal analysis, would suggest a mix composition in the region of:

1 part lime to 17 parts Sand/Silt/clay by volume

with an assessment of the clay to silt/sand content giving a value in the region of

1 part clay to 14.5 parts silty sand.

Sample SR2740-S2 - Original "Clay" Plaster Mortar, *c* 1729

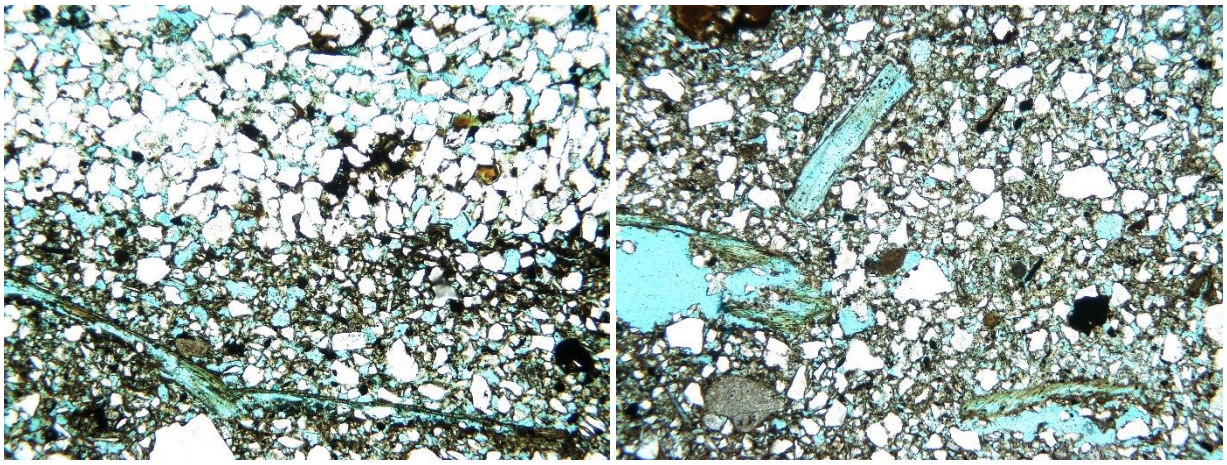
This sample contained several large pieces of a clay plaster, with a quantity of small pieces and some disaggregated fines. The largest fragment measuring 94.5 x 55.0mm in area. The as received moisture content was determined and found to be 1.9% by dry mass.

The intact fragments were found to be finger friable and the lumps could be reduced to powder under moderate to moderately light finger pressure. On examination of the lumps under the stereo-binocular microscope it was noted that the mortar was free of lime inclusions, or large limestone fragments, with no positive reaction observed when droplets of dilute hydrochloric acid were placed onto the mortar surface.

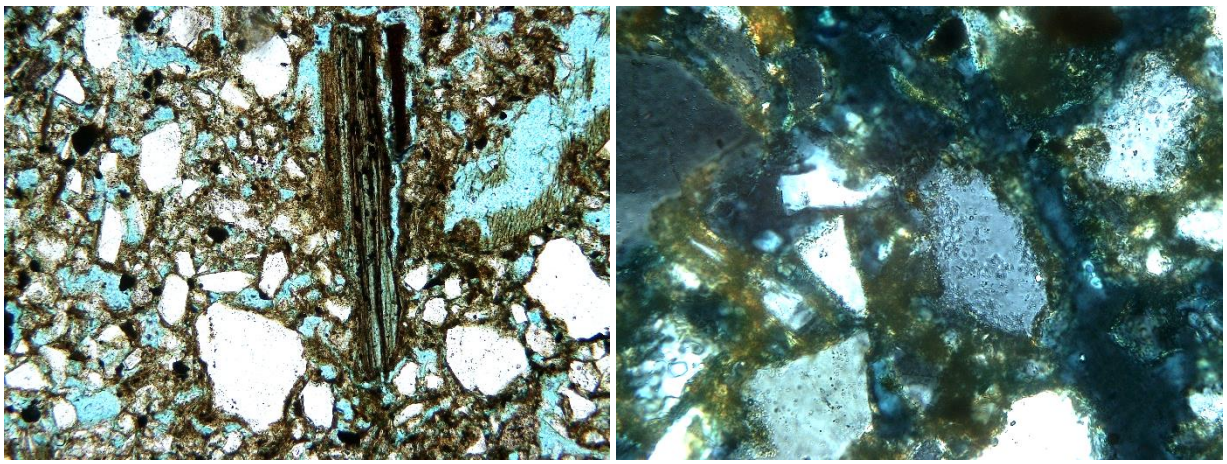
An abundance of straw material was observed within the plaster and it is considered that this was added as a reinforcement, in lieu of animal hair, which, dependent on the geographical area and the status of the building, is not uncommon in clay bound plasters.

Water droplets placed onto the surfaces of intact pieces were rapidly absorbed and diffused through the full thickness of the fragments tested, indicating a high microporosity, with good water and vapour diffusion characteristics.

Plates No. 8, 9 & 10: The left plate shows the sample as received, with the centre image showing an intact piece of plaster, with straw well bound within the clay, with the right plate showing a magnified image of a freshly fractured surface to show the density of straw fibre within the plaster.



Plates No. 11 & 12: The left plate shows a photomicrograph of an area within the mortar containing a large sandstone fragment, which fills the upper part of the plate, with clay bound sand and silt grains in the lower half of the plate. Within the clay bound mortar there is a length of straw, presumably added to the mortar as a reinforcing fibre. The right plate shows another area of mortar where the mortar consists of a clay bound silty sand with an abundance of straw fragments, lower right, upper centre, with a large disrupted fragment in the lower left, the aggregates in view are dominated by quartz grains (white in plate). Both of the above images were recorded under plane polarised light (ppl) with a field of view of 2.4mm.



Plates No. 13 & 14: The left plate was again recorded under ppl, with a field of view of 1.2mm, in this plate the aggregate particles are bound within a clayey silty binder with the grains again dominated by quartz, with small sandstone/siltstone and limestone fragments, with weathered igneous fragments, along with feldspar and muscovite mica. A shard of wood is present in the centre of the plate with a disrupted straw fibre in the upper right (mostly abraded during section preparation and highlighted by blue dyed resin). The plate on the right was recorded under cross polarised light (xpl) with a field of view of 0.64mm, with this showing the dense clay and silt binder encapsulating quartz grains, lithic fragments and feldspar, and was recoded to show that the paste is free of any lime binder (i.e. calcite).

Aggregates in the mortar are dominated by quartz with sandstone and limestone fragments, rare chert, altered igneous rock particles, opaque particles, including both ironstone and coal, along with an abundance of feldspar and mica in the finer fractions. In addition, there is a significant proportion of straw fibre with rare wood fragments present, with rare very localised patches of ash and partially burnt straw which may infer that clay soils were either won from a field after straw burning to clear it of vegetation, or perhaps a proportion of the clay was dried by burning straw and wood to reduce excess moisture.

The results of a point count (modal) analysis to confirm mix composition are presented in the following table:

Sample Ref:	SR2740-S2
Constituents	
Aggregate	**%**
Quartz & chert	35.4
Limestone	3.1
Lithic Fragments	1.7
Sandstone/Siltstone	12.0
Feldspar	4.9
Mica	3.2
ash	0.5
Opaque/Coal/Ironstone	7.8
Straw/wood	6.2
Lime inclusions	-
Total Aggregate	**74.8**
Paste (clay/silt)	25.2
Lime inclusions	0
Clinker	0
Secondary products	0
Total Binder	**25.2**
Total Constituents	**100.0**
Voids & Cracks	**11.0**
	Total
Binder: Aggregate Ratio	**1.0 : 3.0**

Table No. 2: Result of modal analysis on thin section from sample S2

It is not possible from the microscopic examination to differentiate between fine silt fractions and clay minerals and this can be determined by sedimentation and also, to a degree, from the XRD analysis.

Results of Analysis

X-Ray Diffraction

The powdered binder rich sub-samples obtained from each sample were analysed in a Philips X-ray Diffractometer to aid identification of the mineral composition of the binders used in the mortar and plaster production.

For analysis the Diffractometer used was fitted with a single crystal monochromator and set to run over the range 3° to 60° 2θ in steps of 0.1° 2θ at a rate of 1° 2θ/minute using CuKα radiation. With the digital output from the diffractometer analysed by a computer program, which matched the peak positions against the JCPDS International Standard Mineral Data-base sub files using a search window of 0.1°.

The results of the analysis are presented in the following attached Figures, in the form of labelled X-ray Diffractograms:

Figure No. 3: Sample SR2740-S1, Original Bedding Mortar, from a 1729 Limestone built Farmhouse,

Figure No. 4: Sample SR2740-S2, Original Clay Plaster, from a 1729 Limestone Farmhouse.

The abbreviations used on the charts, to identify peak positions, are as follows:

- **cc** = Calcite ($CaCO_3$) calcium carbonate, dominant component of limestone or other calcareous aggregates and from fully carbonated lime binder,
- **qz** = Quartz (SiO_2) natural rock forming mineral, dominant components of the sand in the mortar,
- **fs** = Feldspar, common rock forming minerals, with mostly Albite from the Plagioclase group and Sanidine, an alkali feldspar of the Alkali group,
- **si** = Siderite ($FeCO_3$) Iron Carbonate, Siderite is an iron ore mineral commonly found in hydrothermal veins, and is also a common diagenetic mineral in shales and sandstones,
- **mi** = Muscovite Mica, layer lattice mineral, common rock forming mineral, present as an aggregate component, dominant in the silts and clays,
- **di** = Dickite, clay mineral of the Kaolinite group, formed from the destruction of alkali feldspar.

Quantification by Rietveld Refinement

The results obtained from the XRD analysis were further processed by Rietveld Refinement, to permit quantification, see following:

Sample Reference	**SR2740-S1**	**SR2740-S2**
Component	**% by mass**	
Calcite	15.1	-
Quartz	69.6	79.1
Feldspar – Albite	1.8	5.3
Feldspar – Sanidine	4.2	3.4
Siderite	0.8	-
Muscovite mica	7.9	10.8
Clay mineral – Dickite/Kaolinite	0.6	1.3
Total	**100.0**	**100.0**

On the basis of the XRD analysis, it is indicated that both samples are clay mortars, although the actual clay mineral content is low. Sample S1 from the masonry bedding mortar contains a clay binder with a proportion of lime, and limestone dust (from the microscopic examination) whereas sample S2 is a clay mortar with no lime or limestone present in the sample analysed.

The clays in both samples are dominated by Dickite, a Kaolinite clay. The aggregates, in both samples is dominated by quartz, with muscovite mica and feldspar minerals present. Sample S1 also contains Siderite and this is likely to have been incorporated with the aggregate as an ore mineral, ironstone or within the sandstone fragments or possibly associated with the limestone in this sample.

Mix Composition

Sample SR2740-S1 - Original "Clay" Bedding Mortar, *c* 1729

Results of the mix composition as determined by Acid Digestion

The result of the composition analysis, determined by acid digestion, corrected for the presence of limestone fragments in the sample, as determined from the modal analysis, are presented below:

Sample Ref. No.	**SR2740-S1**
Description:	**Bedding Mortar**
Binder Type (from XRD)	Non Hydraulic Lime
Binder form (from Microscopic examination)	Quicklime
Weight mix ratio calculated by dry mass:	
Lime	1.0
Clay/Sand/Aggregate mix	40.3
Approximate Volume Proportions calculated on the basis of the standard data for a Non-Hydraulic Quicklime:	
Lime	1.0
Clay/Sand/Aggregate mix	16.9

The aggregate residue, remaining after acid digestion, was recovered and the particle size distribution determined, with the results presented below and as an aggregate filled histogram in figure No. 1.

Sample Reference	**SR2740 – S11**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	0	100
2.00mm	0.1	99.9
1.00mm	0.3	99.6
0.500mm	0.8	98.8
0.250mm	2.7	96.1
0.125mm	44.8	51.3
0.063mm	22.7	28.6
Passing	28.6	

Table No. 1: Grading of aggregate recovered following acid digestion of Sample S1.

Aggregates in the sample are dominated by quartz, with a minor proportion of chert, silty sandstone fragments, ironstone, limestone fragments, with both Oolitic and Bioclastic (containing gastropods, forams and indeterminate shell fragments) forms present, in addition feldspar, mica and indeterminate clay minerals are present within the fines.

On the basis of the mineral composition of the sand, the particle size distribution and the sub-rounded to sub-angular particle shape, the ‘clay’ mortars are likely to have been obtained as an-as dug silty clay material, perhaps enriched with sand from a local watercourse or river terrace. With a low proportion of lime added to either dry the mortar, during mixing, or to contribute to the strength of the mortar as placed.

Sample SR2740-S2 - Original "Clay" Plaster Mortar, *c* 1729

Results of the mix composition as determined by Wash Grading

From the examination of sample S2 it is indicated that the mortar used in the plaster was a clay mortar, with no evidence of any lime having been added. As there was no lime present the composition of the mortar was determined from a particle size distribution. With the grading determined by wash grading the mortar over a 32µm sieve, with sedimentation of the waters passing the sieve.

The results of the particle size distribution are presented in the table below and in aggregate filled histogram in figure No. 2.

Sample Reference	**SR2740 – S2 Clay Plaster**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	0.7	99.3
2.00mm	0.3	99.0
1.00mm	0.5	98.5
0.500mm	0.7	97.8
0.250mm	1.9	95.9
0.125mm	9.0	86.9
0.063mm	26.0	60.9
0.032mm	36.2	24.7
Passing	24.7	

Silt and Clay content was determined by simple sedimentation and found to be in the region of:

	% of Sand/Clay/Silt fraction by mass	
Sand Content (>0.063mm)	39.1	
Silt Content (<0.063mm >0.004mm)	55.8	
Clay Content (<0.004mm)	5.1	(clay + Fine Silt = 24.7%)

The sand in sample S2 is dominated by quartz along with a proportion of fine grained sandstone and Limestone fragments, altered (weathered) igneous rock fragments, with feldspar and muscovite mica in the silt fractions and it is likely that it was obtained from the same location as that used as the source for the materials in sample S1 (Building mortar).

Summary

From the examination and analysis of the samples of the bedding mortar and plaster received, it is indicated from the analysis that they are both different, in that the bedding mortar contained a proportion of lime, whilst the plaster is a clay mortar with no lime.

However, the aggregates all show some similarity in the minerals present, and their relative proportions, and are likely to be from the same location/geographical area, though the minor variations may again suggest either a separation in time, or level within the deposit worked.

Details of the mixes sampled from the 1729 Farmhouse building are summarized below:

Sample Ref. No.	**SR 2740-S1 Bedding Mortar**	**SR2740-S2 Plaster**
Binder type (from XRD)	Clay with High Calcium Lime	Silty Clay
Binder form:	As Dug Sand/Silt/Clay + Quicklime	As-Dug Sand/Silt/Clay
Approximate volume Proportions calculated on the basis of the standard data for each binder type.		
Lime	1.0	
Clay + Silt	4.9	1.0
Aggregate	12.0	18.6
Mix Composition from Modal Analysis (visual volume proportions)		
Clay + Fine silt		1.0
Lime + Clay + Fine silt	1.0	
Aggregate (Total)	1.4	3.0
Aggregate (Effective)	1.6	

Sample Reference	**SR2740 – S1**		**SR2740 – S2**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100	0	100
4.00mm	0	100	0.7	99.3
2.00mm	0.1	99.9	0.3	99.0
1.00mm	0.3	99.6	0.5	98.5
0.500mm	0.8	98.8	0.7	97.8
0.250mm	2.7	96.1	1.9	95.9
0.125mm	44.8	51.3	9.0	86.9
0.063mm	22.7	28.6	26.0	60.9
0.032mm			36.2	24.7
Passing	28.6		24.7	

Quality Statement

We confirm that in the preparation of this report we have exercised reasonable skill and care.

The results presented, and comments offered relate only to the samples submitted to CMC by Nigel Copsey of the Earth, Stone & Lime Company, on the 18th July 2019, which were identified as bedding mortar and plaster samples from a 1729 Limestone Farmhouse in Malton, North Yorkshire.

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For CMC Ltd.

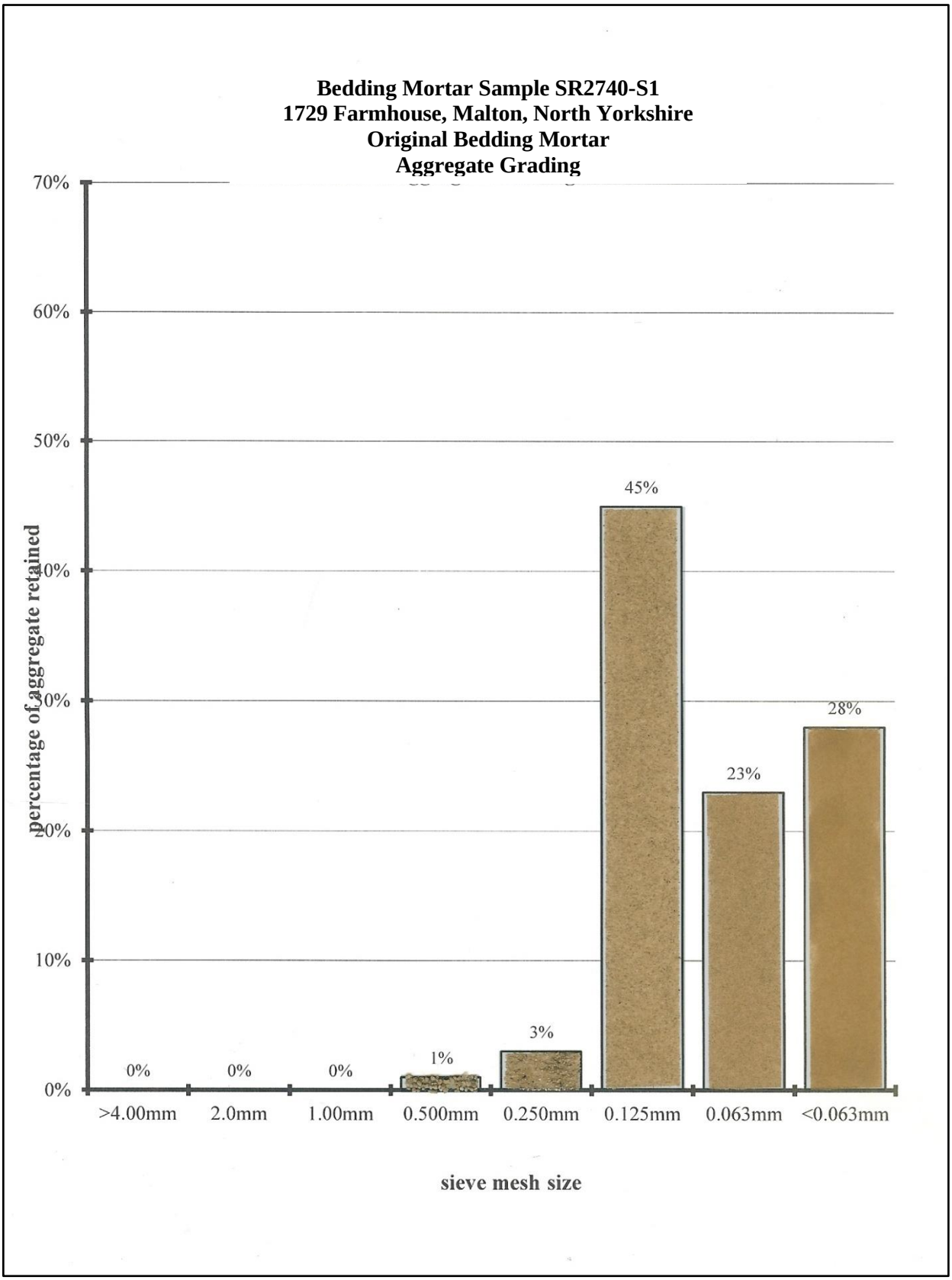

Figure No. 1 – Grading of recovered aggregate from Sample SR2740-S1

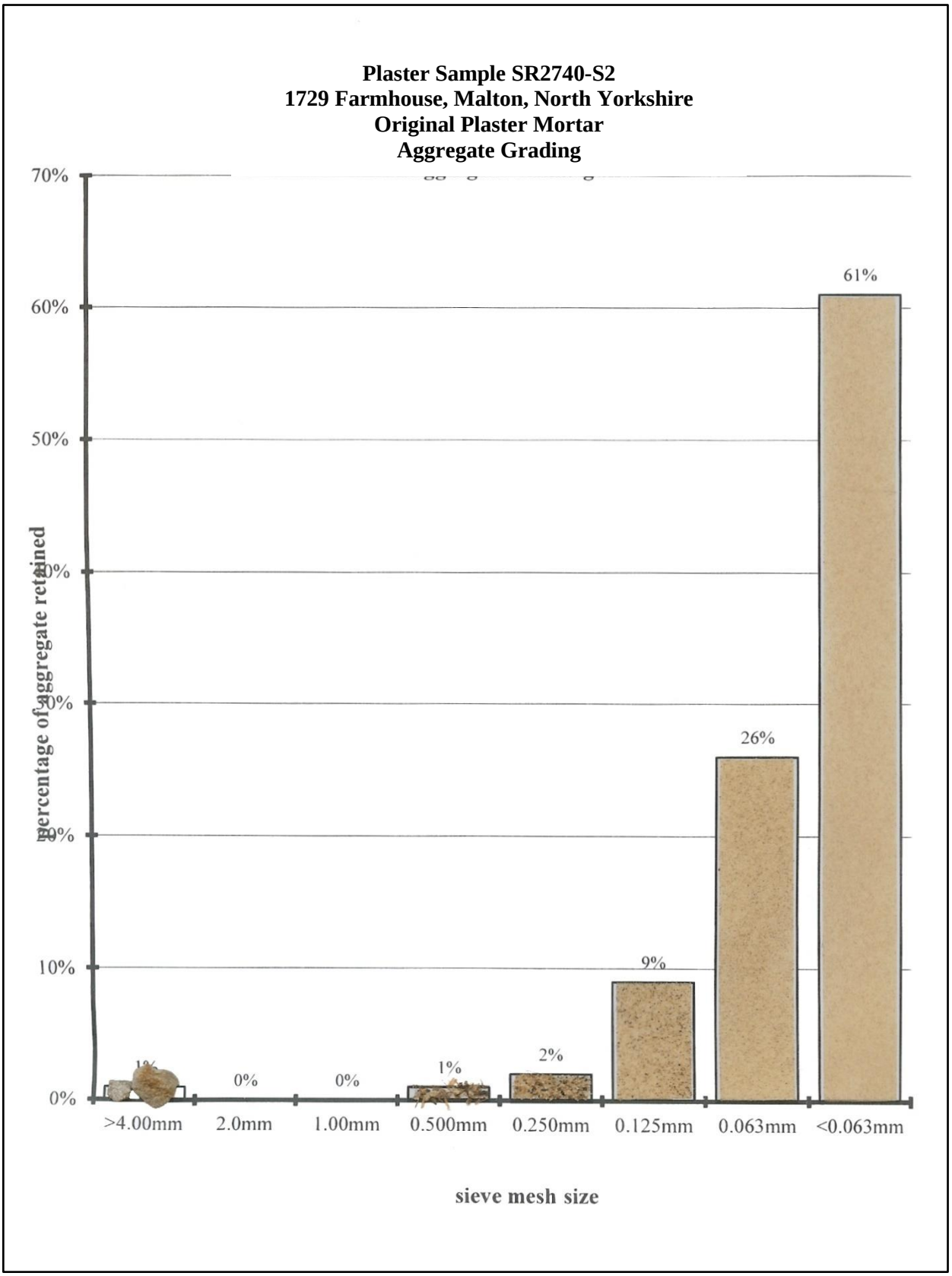

Figure No. 2 – Grading of recovered aggregate from Sample SR2740-S2

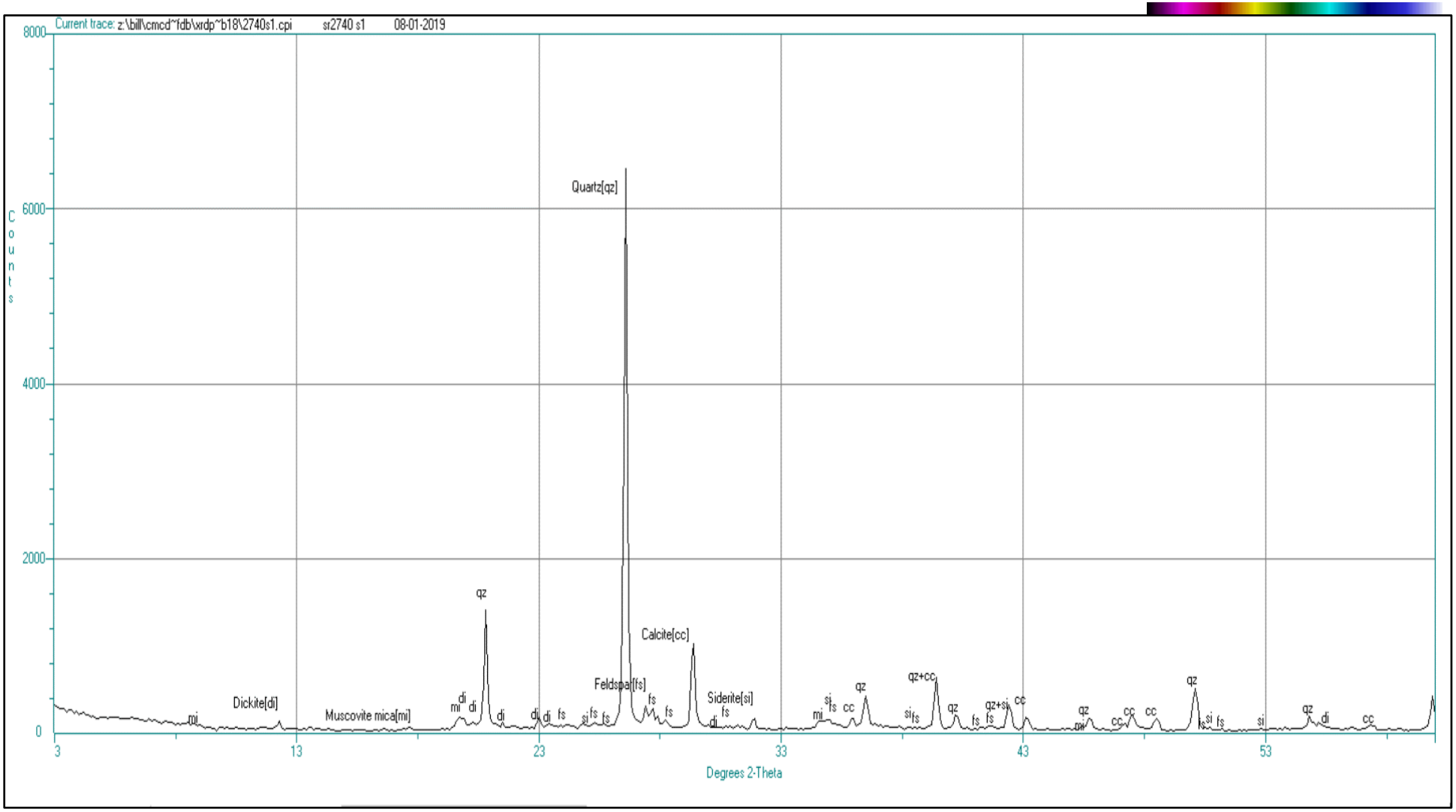

Figure No. 3: Sample SR2740-S1, Original Bedding Mortar, from a 1729 Limestone built Farmhouse.

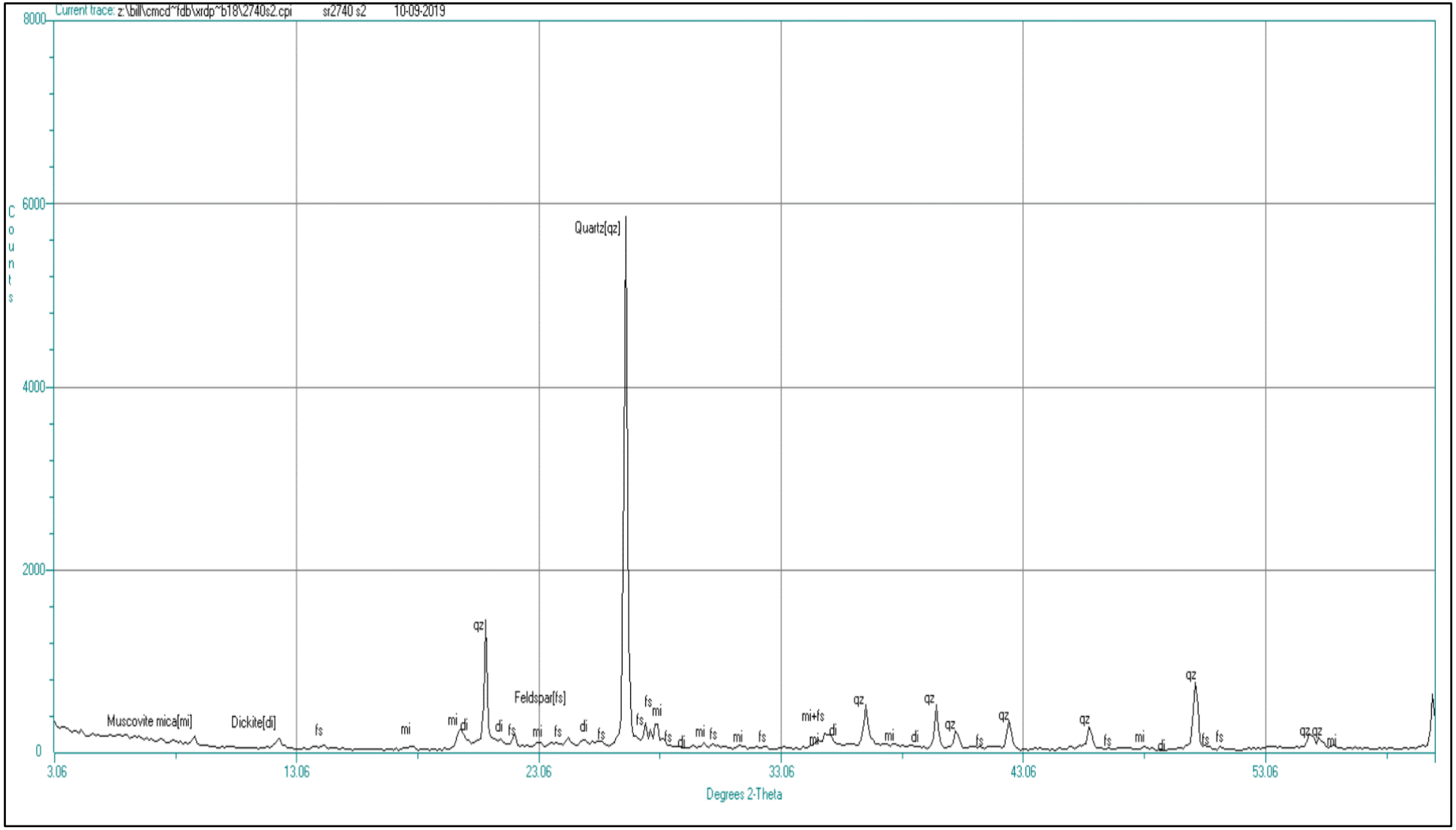

Figure No. 4: Sample SR2740-S2, Original Clay Plaster, from a 1729 Limestone Farmhouse.